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(पहला पुनरीक्षण)

Indian Standard

DRIED MILK — DETERMINATION OF MOISTURE CONTENT (REFERENCE METHOD)

(First Revision)

ICS 67.100.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 2008

Price Group 4

Dairy Products and Equipment Sectional Committee, FAD 19

NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical with ISO 5537 : 2004 'Dried milk — Determination of moisture content (Reference method)' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dairy Products and Equipment Sectional Committee and approval of the Food and Agriculture Division Council.

This standard was first published in 1986 based on the earlier version of International Dairy Federation (IDF) Standard. Subsequently to align with the latest ISO and IDF Standards on the subject which are identical, this standard is being revised under dual numbering with ISO 5537 : 2004.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

DRIED MILK — DETERMINATION OF MOISTURE CONTENT (REFERENCE METHOD)

(First Revision)

WARNING — The use of ISO 5537|IDF 26 may involve the use of hazardous materials, operations, and equipment. This standard does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of local regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the moisture content of all types of dried milk.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
moisture content
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The moisture content is expressed as a percentage by mass.

3 Principle

A test portion is dried in a drying oven set at 87 °C for 5 h while dry air is passed through the test portion. The loss of mass of the test portion (which is related to the content of "non-chemically bound" water) is determined.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

4.2 Drying oven, capable of being maintained at 87 °C ± 1 °C throughout the working space, with forced ventilation, thermostatically controlled, with the following equipment (see also Figure A.1).

4.2.1 Metal block, provided with channels of diameter 4,3 mm for holding the columns (4.4) in the drying oven.

4.2.2 Copper tubes, of length 1 500 mm, of internal diameter 2 mm, connected to the metal block in the drying oven.

4.2.3 Constant pressure regulator, provided with restrictors, capable of delivering 33 ml/min of dry air to each column in the drying oven.

4.2.4 Tube, made of polycarbonate, of length 350 mm, of diameter 40 mm, filled with silica gel with hygrometric indicator.

The silica gel should have been dried at 150 °C for more than 12 h before use. Using the dry compressed air (4.11), no colour change of the hygrometric indicator should be noticed.

4.3 Desiccator, containing freshly dried silica gel with hygrometric indicator.

4.4 Columns, made of hard polypropylene (Phenomenex 1213–10211)[1], of length 90 mm, of internal diameter 20 mm, provided with two polyethene filters (Phenomenex 1212--1023)[1], narrowed towards one end to fit onto the block (4.2.1).

4.5 Synthetic stoppers, made of soft polyethylene (Emergo 20273 B198 and 20371 U1)[1].

4.6 Container, suitable for holding the columns (4.4).

4.7 Container, suitable for holding the synthetic stoppers (4.5).

4.8 Rod, made of polyvinyl chloride (PVC), of length 120 mm, of diameter 18 mm, suitable for placing the polyethylene filters in the column (4.4).

4.9 Tweezers, suitable for removing the polyethylene filters from the column (4.4).

4.10 Soap-film meter, suitable for measuring a flow of 33 ml/min.

4.11 Dry compressed air, minimum pressure of 200 kPa, moisture content of $\leqslant$ 0,01 mgH_2O per litre at atmospheric pressure, free of any organic material. Use metal tubes only to connect the source of compressed air to the equipment in the drying oven (4.2).

4.12 Container, made of glass, provided with an airtight lid.

NOTE The apparatus mentioned in 4.2 and those in 4.4 to 4.8 are available commercially (e.g. Elbanton and Funke Gerber)[2].

5 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

6 Preparation of test sample

Transfer the whole test sample to a dry, hermetically closed container (4.12) of capacity approximately twice the volume of the sample. Mix intensively by rotating and shaking the container.

Use a statistical sampling plan if there is evidence of sample inhomogeneity even after the intensive mixing mentioned above.

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1) Phenomenex and Emergo are examples of suitable products available commercially.

2) Elbanton and Funke Gerber are the trade names of products supplied by Elbanton b.v., Uitingstraat 18, 5331 EJ, Kerkdriel (NL) and Funke-Dr.N.Gerber Labortechnik GmbH, Ringstrasse 42, 12105, Berlin, respectively.

The information in footnotes 1) and 2) is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO or IDF of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7 Procedure

7.1 Preparation of column

7.1.1 Set the constant pressure regulator at approximately 100 kPa. Measure, using the soap-film meter (4.10), the airflow of each channel. Calculate the average flow per channel. Adjust the pressure, if necessary, to obtain an average airflow of 33 ml/min per channel.

7.1.2 Remove both synthetic stoppers from the column (4.4). Place the stoppers in the container (4.7) and store at room temperature.

7.1.3 Place the column, provided with filters placed in position as shown in Figure A.1, in the metal block (4.2.1) in the oven (4.2) set at 87 °C for at least 1 h.

Use slight pressure when placing the column in position to create an airtight connection.

7.1.4 Take the column out of the oven and close it with the synthetic stoppers (see 7.1.2). Place the closed column in the container (4.6) with the other prepared columns. Place the container and the columns in the desiccator (4.3). Close the desiccator and allow the whole to cool for 60 min ± 5 min.

7.2 Preparation of test portion

7.2.1 After cooling (see 7.1.4), take one closed column at a time out of the container while leaving the container in the desiccator. Close the desiccator immediately after taking out a column. Weigh the closed column to the nearest 1 mg, recording the mass to four decimal places.

7.2.2 Remove the synthetic stoppers from the pre-weighed column (see 7.2.1). Using tweezers (4.9), also remove the upper filter from the column. Keep the stoppers and the filter in a dry place in the weighing room.

7.2.3 Add 5,0 g ± 0,3 g of prepared test sample (Clause 6) to the column. Using the rod (4.8), place the upper filter back in position in the column. Remove any dried milk above the filter with a clean tissue. Close the column with both synthetic stoppers (see 7.2.2).

7.2.4 Immediately weigh the closed column to the nearest 1 mg, recording the mass to four decimal places. Open the desiccator, place the closed column back into the container and close it again.

7.2.5 When the analysis involves more than one test sample, prepare all test portions by repeating the procedure from 7.2.1 to 7.2.4 for each separate test portion. Handle only one column at a time.

7.3 Determination

7.3.1 Open the desiccator. Take one closed column with the prepared test portion (see 7.2.4) at a time out of the container. Remove both synthetic stoppers from each column. Place the stoppers in the container (4.7) and store at room temperature.

7.3.2 Place each column and its contents in the metal block (4.2.1) which is placed in the drying oven (4.2). As soon as ready, close the oven. Dry the columns and their contents in the oven (4.2) set at 87 °C for 5 h.

7.3.3 After drying, remove each column from the metal block. Replace both synthetic stoppers. Open the desiccator and place the dried columns and their contents back into the container (4.6). Immediately close the desiccator after placing the last column into the container. Allow the whole to cool for 60 min ± 5 min.

7.3.4 After cooling (see 7.3.3), open the desiccator and, in case of more than one test sample, take one closed column at a time out of the container leaving the container in the desiccator. Close the desiccator immediately after taking out a column. Weigh the closed column to the nearest 1 mg, recording the mass to four decimal places.

8 Calculation and expression of results

8.1 Calculation

Calculate the mass fraction of moisture in the sample, w, using the following equation:

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100\ \%$$

where

m_0 is the numerical value of the mass of the column, filters and stoppers (see 7.2.1), in grams;

m_1 is the numerical value of the mass of test portion, the column, filters and stoppers before drying (see 7.2.4), in grams;

m_2 is the numerical value of the mass of the test portion, column, filters and stoppers after drying (see 7.3.4), in grams.

8.2 Expression of test results

Express the test results to two decimal places.

9 Precision

9.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in Annex B. The values derived from this test may not be applicable to concentration ranges and matrices other than those given

9.2 Repeatability

The absolute difference between two individual single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,15 %.

9.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,20 %.

10 Test report

The test report shall specify:

a) all information required for the complete identification of the sample;

b) the sampling method used, if known;

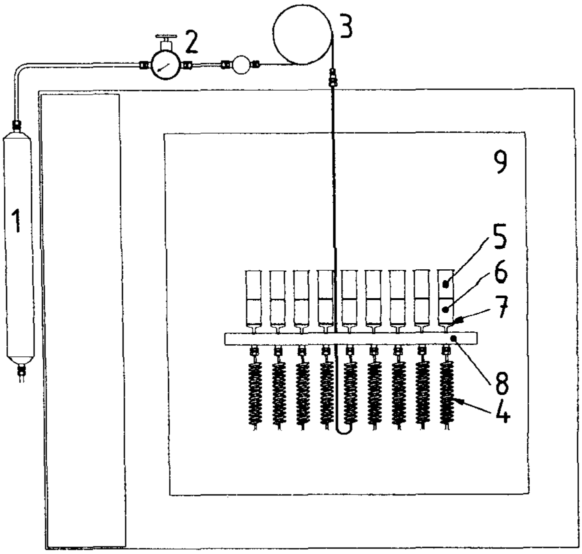
c) the test method used, together with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained or, if the repeatability has been checked, the final quoted results obtained.

Annex A
(informative)

Drying apparatus



Key

1 tube of polycarbonate
2 constant pressure regulator
3 restrictor
4 copper tube
5 filter of polyethene
6 container
7 filter of polyethene
8 metal block
9 drying oven

NOTE 1 Every hard polypropylene column in the metal block is separately connected to a copper tube (inside oven length is 1,5 cm). Outside the oven, every copper tube is connected to a restrictor with an inlet pressure of approximately 1 bar.

NOTE 2 During the drying process, dry air is heated in the drying oven through a copper tube (of length 1,5 m and of internal diameter 2 mm) and then passed through the columns with dried milk.

Figure A.1 — Apparatus for determination of the moisture content of dried milk

Annex B
(informative)

Results of interlaboratory test

An international collaborative test (see [5]) involving eight laboratories was carried out on eight samples of whole milk powder (WMP) and skimmed milk powder (SMP), obtained from Austria (1), Finland (2) and Spain (3) respectively. The levels of the moisture content in the samples varied from a mass fraction of 2,38 % to 3,93 %.

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Table B.1.

Table B.1 — Results of the interlaboratory test

	SMP (1)	SMP (2)	SMP (3)	WMP (1)	WMP (2)	WMP. (3)
Number of participating laboratories after eliminating outliers	8	8	8	8	8	8
Mean value, %	3,62	3,57	3,93	2,52	3,16	2,38
Repeatability standard deviation, s_r, %	0,052	0,085	0,053	0,045	0,035	0,049
Coefficient of variation of repeatability, %	1,44	2,38	1,34	1,80	1,11	2,06
Repeatability limit, r, (2,8 s_r), %	0,146	0,238	0,148	0,126	0,084	0,137
Reproducibility standard deviation, s_R, %	0,058	0,096	0,074	0,055	0,060	0,098
Coefficient of variation of reproducibility, %	1,61	2,69	1,89	2,19	1,89	4,11
Reproducibility limit, R (2,8 s_R), %	0,162	0,296	0,207	0,154	0,168	0,274

Bibliography

[1] ISO 707[3], *Milk and milk products — Guidance on sampling*

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

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[5] DE KNEGT, R.J. and BRINK, H. v.d. Improvement of the Drying Oven Method for the Determination of the Moisture Content of Milk Powder, *Int. Dairy Journal*, **8**, 1998, pp. 733–738

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3) Equivalent to IDF 50.

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